

## SLOVENSKI STANDARD oSIST prEN ISO 11357-8:2020

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Polimerni materiali - Diferenčna dinamična kalorimetrija (DSC) - 8. del: Ugotavljanje toplotne prevodnosti (ISO/DIS 11357-8:2019)

Plastics - Differential scanning calorimetry (DSC) - Part 8: Determination of thermal conductivity (ISO/DIS 11357-8:2019)

Kunststoffe - Dynamische Differenz-Thermoanalyse (DSC) - Teil 8: Bestimmung der Wärmeleitfähigkeit (ISO/DIS 11357-8:2019)

Plastiques - Analyse calorimétrique différentielle (DSC) - Partie 8: Détermination de la conductivité thermique (ISO/DIS 11357-8:2019)

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### Plastics — Differential scanning calorimetry —

Part 8:

**Determination of thermal conductivity** 

ICS: 83.080.01

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#### **Foreword**

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A list of all parts in the ISO 11357 series can be found on the ISO website. 8-44d9-475a-bba5-

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#### Introduction

The advantage of using DSC for measuring the thermal conductivity of plastics is that with the same instrument also the specific heat capacity can be obtained. This enables the determination of the thermal diffusivity by dividing the thermal conductivity by the density and specific heat capacity.

In addition, DSC instruments are widely-used and available in almost all test institutes and labs. Hence, measurements of thermal conductivity can be done without need for procurement of an additional instrument.

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### Plastics — Differential scanning calorimetry —

### Part 8:

### **Determination of thermal conductivity**

#### 1 Scope

This document establishes a method for determination of the thermal conductivity of solid unfilled and filled or fibre reinforced plastics and composites by means of Differential Scanning Calorimetry (DSC).

It is applicable for materials with thermal conductivities of up to  $1 \text{ W/(m \cdot K)}$ .

#### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 291, Plastics — Standard atmospheres for conditioning and testing

ISO 472, Plastics — Vocabulary

ISO 6344-1, Coated abrasives — Grain size analysis — Part 1: Grain size distribution test

ISO 11357-1, Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles

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#### 3 Terms and definitions d151f83a/sist-en-iso-11357-8-2021

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 11357-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at <a href="http://www.electropedia.org/">http://www.electropedia.org/</a>

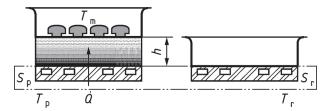
#### 4 Principle

For the determination of thermal conductivity, the usual placement of the specimen in the sample holder position is modified according to a procedure proposed in the literature.[1],[2] Additional details on scientific background, deduction of results and performance of measurements can be found in[3] and [4].

As usual, an empty crucible is placed in the reference position of the sample holder assembly. The test specimen is placed directly onto the sensor of the sample position and a crucible containing a substance of known melting temperature is put on top of the specimen (see Figure 1). The thermal conductivity is measured at a temperature slightly above the melting point of this substance in the small temperature range in which the slope of the melting peak with test specimen is determined (see 9.2, Figure 2)..

Upon heating, a temperature gradient is created in the specimen. The temperature of the top of the specimen remains constant at the melting temperature  $T_{\rm m}$  of the melting substance while the temperature of the bottom side of the specimen corresponds to the temperature of the sample side sensor. The differential heat flow between sample and reference sensor is measured by the DSC instrument.

From the temperature difference between top and bottom of the sample  $(T_{\rm m} - T_{\rm p})$ , the sample height h and the heat flow rate difference  $\dot{Q}$  between sample and the reference sensor, the thermal conductivity of the specimen is determined.



#### Key

- *h* height of specimen
- $\dot{Q}^{-}$  heat flow rate difference between sample and reference position
- S<sub>p</sub> sensor of sample position
- S<sub>r</sub> sensor of reference position
- $T_{
  m m}$  melting temperature of known substance and temperature of top of specimen, respectively
- $T_{\rm p}$  temperature of sample position and bottom side of specimen, respectively
- $T_{\rm r}$  temperature of reference position

Figure 1 — Schematic layout of measurement

### 5 Apparatus and substances standards.iteh.ai)

#### 5.1 DSC instrument

A DSC instrument in accordance with specifications given in ISO 11357-1 shall be used. ba5-

Only original raw data shall be used without any further data processing.

NOTE According to currently available results the method has been verified with heat-flux type DSC instruments only. However, it is expected that power-compensated DSC instruments are suitable, too.

#### 5.2 Crucibles

Aluminium crucibles shall be used fitting the DSC instrument. Both, open or ventilated closed crucibles are suitable. Upon using sealed crucibles suitable precautions shall be taken to prevent excessive inside pressure.

Care shall be taken to avoid deformation of the plane crucible bottom.

NOTE Uneven crucible bottoms may result in too low thermal conductivity values.

#### 5.3 Melting substance

Certified melting substances or melting substances having a purity of 99,9999 % or better shall be used.

The melting temperature determines the temperature of measurement of the thermal conductivity.

Melting temperatures of specimens to be measured shall be above those of the melting substances to be used. In case of doubt, this shall be ensured by performing suitable preliminary tests.

NOTE Metals such as gallium or indium have been found suitable.

#### 5.4 Protective lacquer

If metals that can react with the crucible material are used as melting substances, protective lacquers can be applied for coating the crucible inside surface and preventing formation of alloys with the aluminium crucibles.

NOTE 1 Gallium, for example, is known to form alloys with aluminium.

The protective coating shall be thermally stable in the measurement temperature range and shall be applied continuously on the crucible inside surface in constant thickness of not more than  $30 \mu m$ .

Both, sample and reference crucible shall be coated in exact the same manner.

NOTE 2 Suitable examples for protective lacquers are e.g. nitro lacquer or permanent marker ink.

#### 5.5 Length measuring device

Suitable device for measuring the diameter with an accuracy of  $\pm 20~\mu m$  or better and the specimen height with an accuracy of  $\pm 10~\mu m$  or better.

#### 6 Specimens

#### 6.1 Geometry

Cylindrical specimens shall be used.

The diameter shall be measured with an accuracy of  $\pm 20~\mu m$  or better and shall not deviate from the diameter of the bottom of the crucible by more than 0,1 mm. The height shall be measured with an accuracy of  $\pm 10~\mu m$  or better and shall be in the range of 0,5 mm to 2,0 mm.

To ensure the best possible thermal contact to the sensor and crucible, specimens shall be free of scars and burrs and shall have plane-parallel upper and lower surfaces. It is recommended to grind the upper and lower surface using ultra-fine abrasive paper of grit size P800 in accordance with the ISO/FEPA scale specified in ISO 6344-1or better.

NOTE Higher surface roughness may result in reduced thermal contact and lower values of thermal conductivity.

#### 6.2 Number and sampling

At least five specimens shall be measured and the results averaged.

For homogeneous filled and unfilled materials no particular sampling procedures are required.

For fibre reinforced laminates and composites specimens shall be taken at least 10 mm away from sheet edges. Specimens shall be taken from several homogeneously distributed locations of the sheet with exclusion of defective areas.

NOTE Depending on sample preparation and for samples with anisotropic reinforcements the thermal conductivity can depend on the direction of measurement. In such cases the direction of measurement should be noted.

#### 7 Conditioning

Prior to measurement specimens shall be conditioned for a minimum of 2 h in accordance with ISO 291.

Moisture sensitive materials shall be dried preferably in accordance with relevant material standards. Alternatively, drying conditions shall be agreed between involved parties.